Electronic Supplementary Material (ESI) for New Journal of Chemistry.

Supporting Information

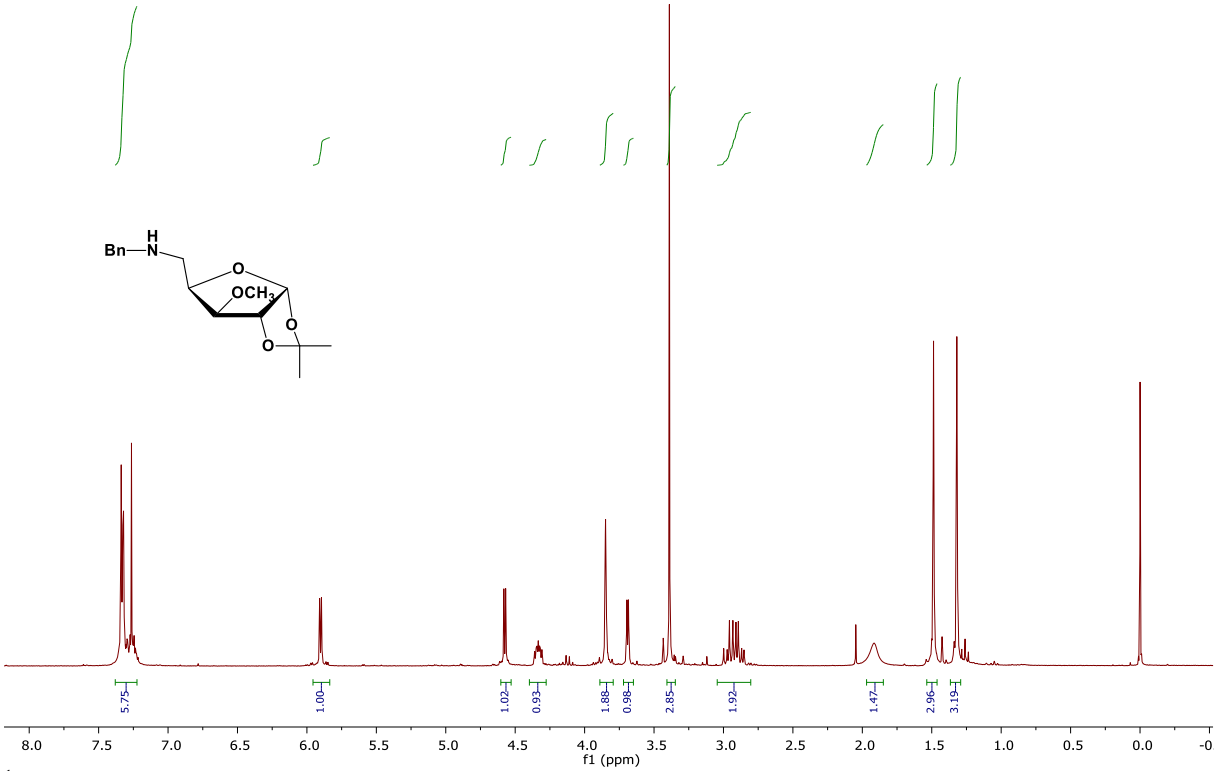
The conformations of new CF_3 and CF_3-CHF containing amides derived from carbohydrates: NMR, crystallographic and DFT study

Monika Bilska-Markowska*, Tomasz Siodła, Ewa Patyk-Kaźmierczak, Andrzej Katrusiak, Henryk Koroniak

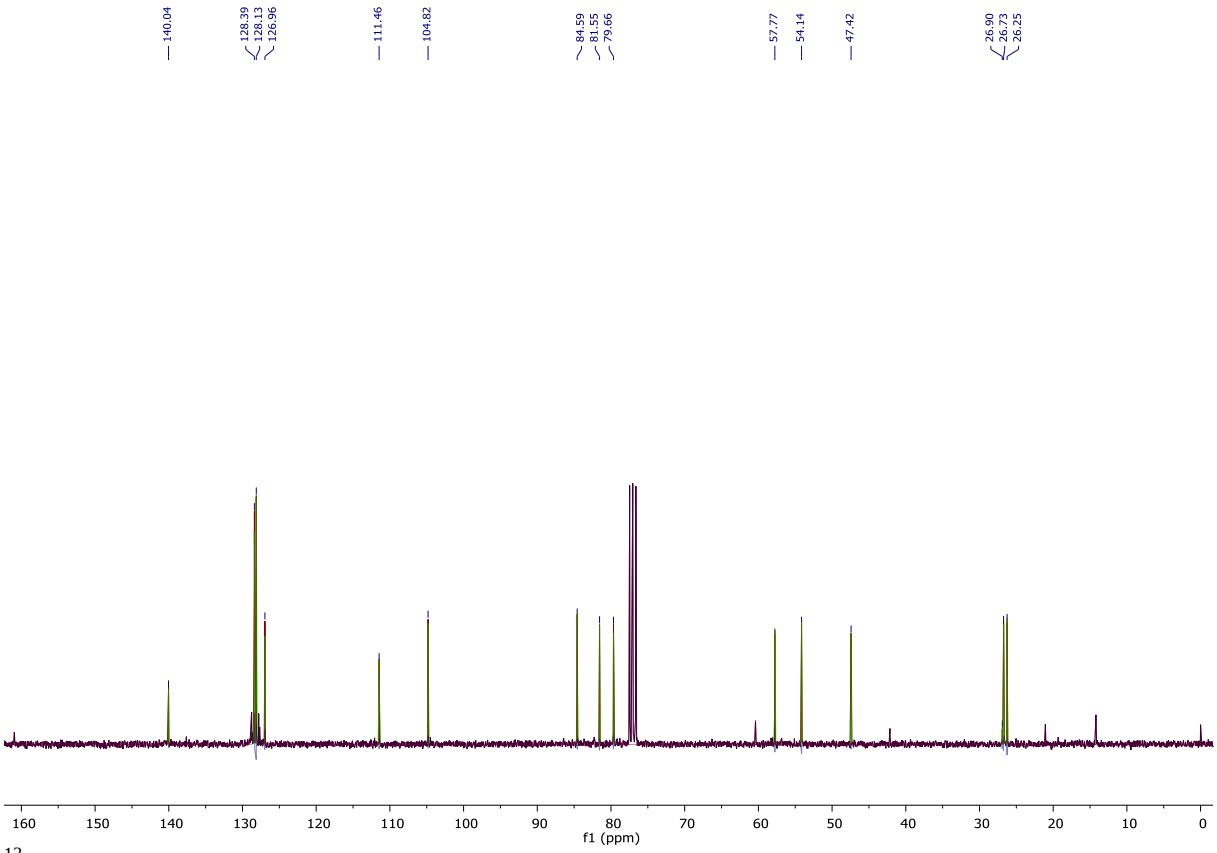
Faculty of Chemistry, Adam Mickiewicz University, Umultowska 89b, 60614 Poznan (Poland)
E-mail: mbilska@amu.edu.pl

I) ^{1}H, ^{13}C, ^{19}F NMR Spectra of Compounds

1) Compound **1a**

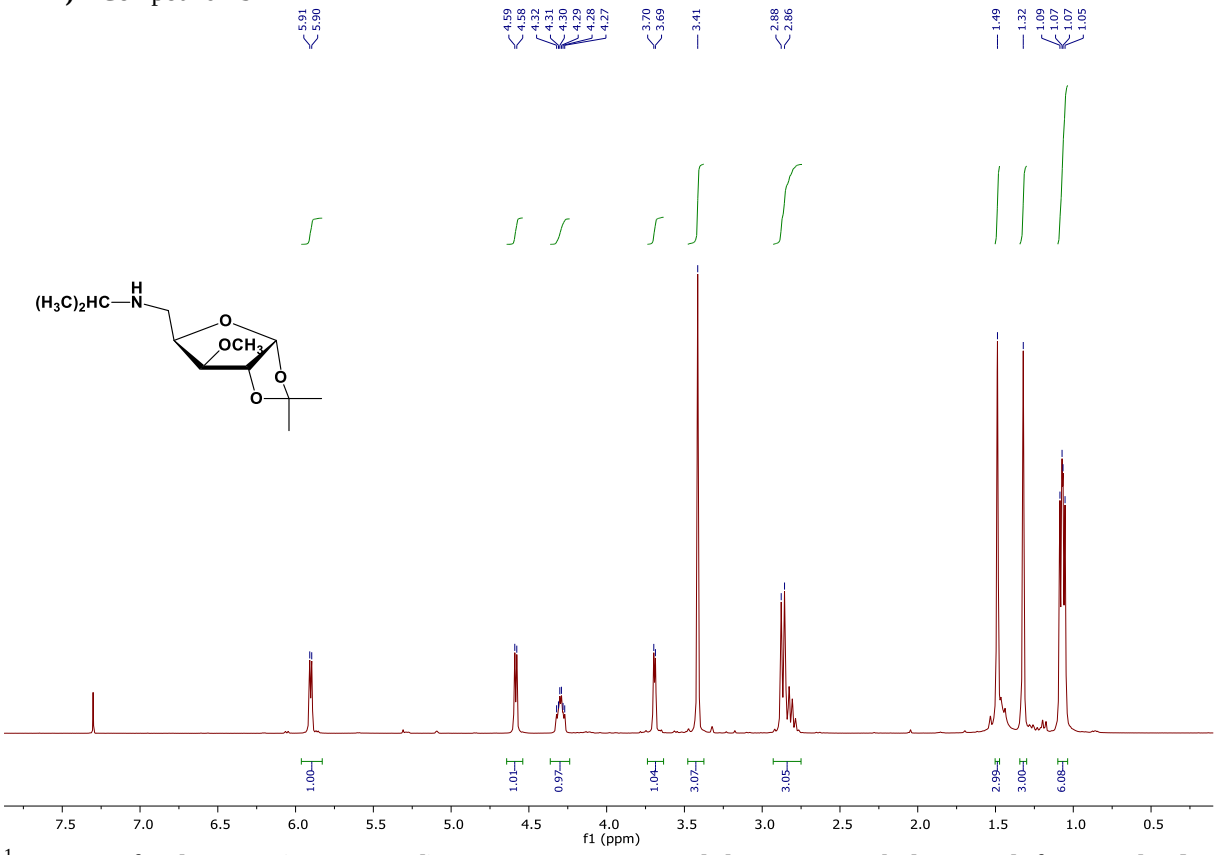


^{1}H NMR of 5-(*N*-benzyl)amino-5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-α-D-xylofuranoside **1a**

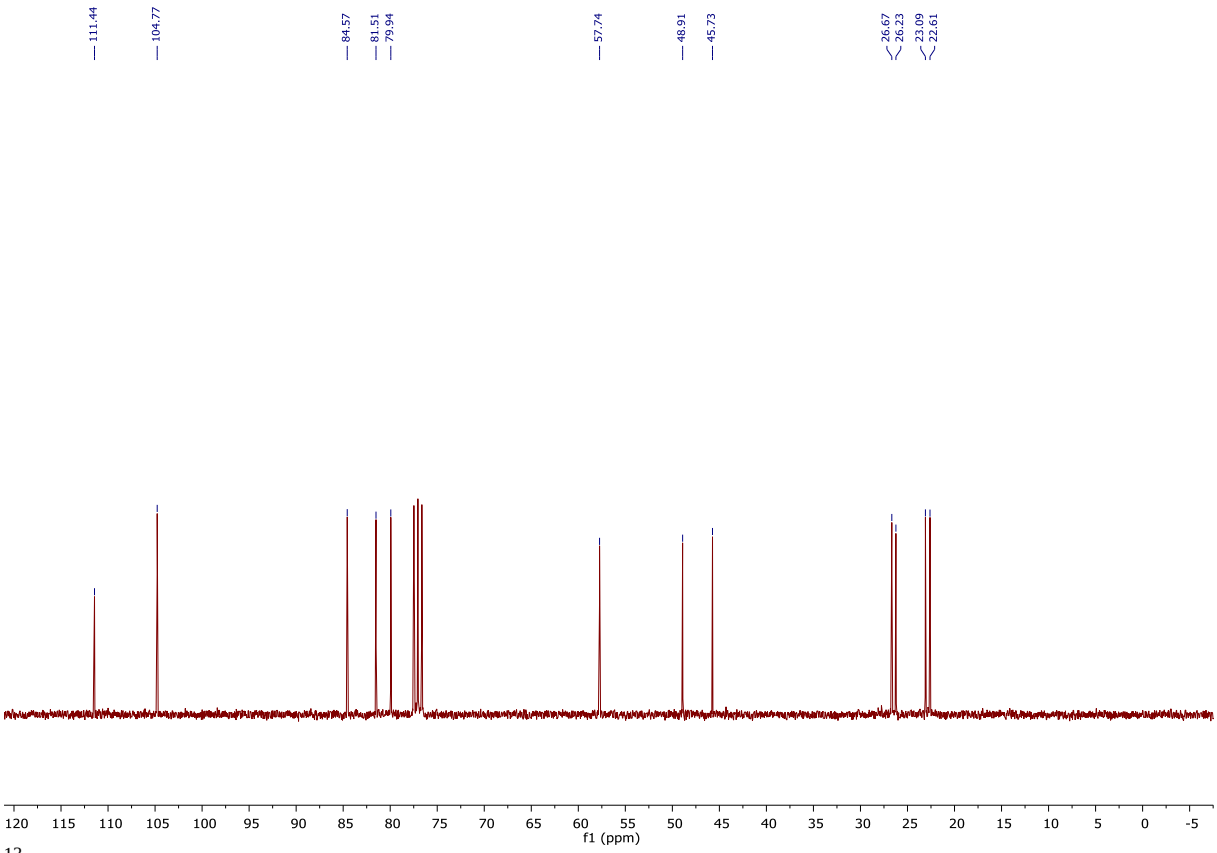


^{13}C NMR of 5-(*N*-benzyl)amino-5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-α-D-xylofuranoside **1a**

2) Compound **1b**

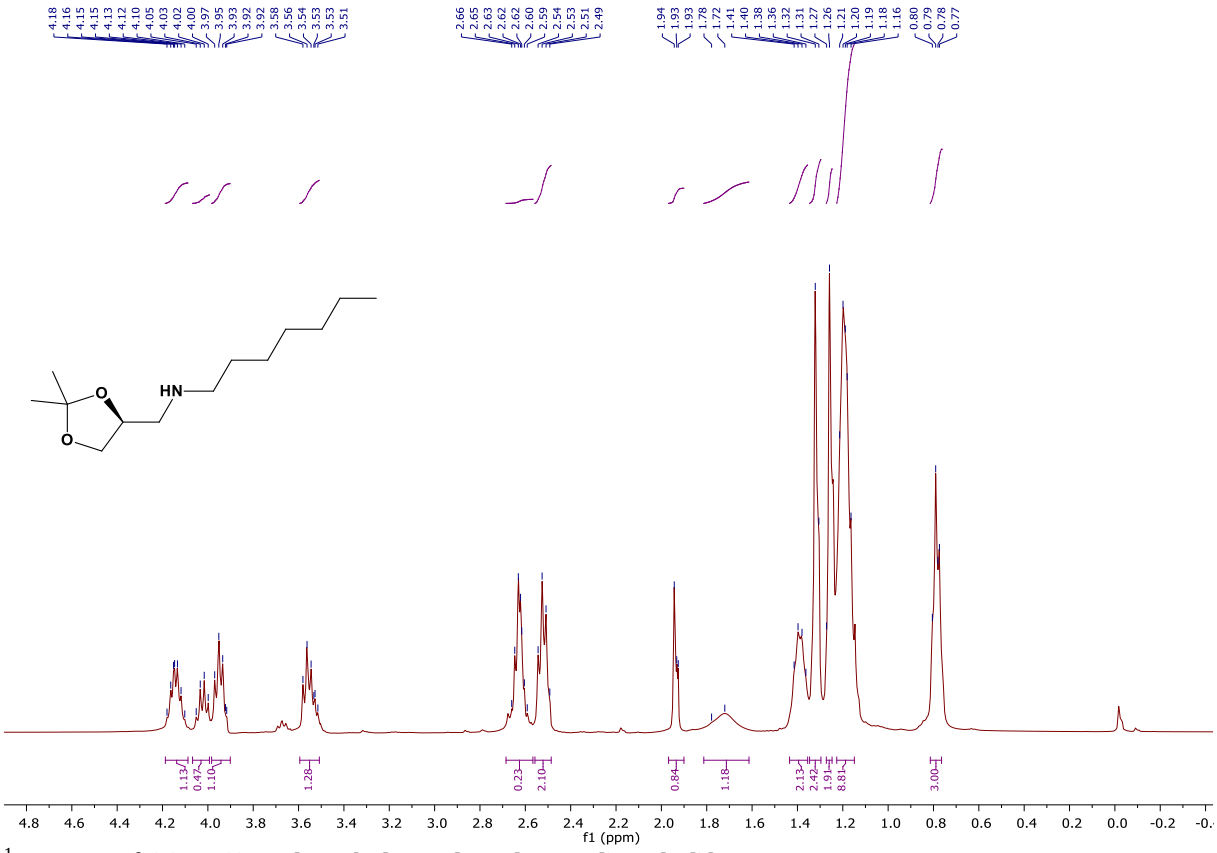


^{1}H NMR of 5-deoxy-5-(*N*-isopropyl)amino-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranoside **1b**

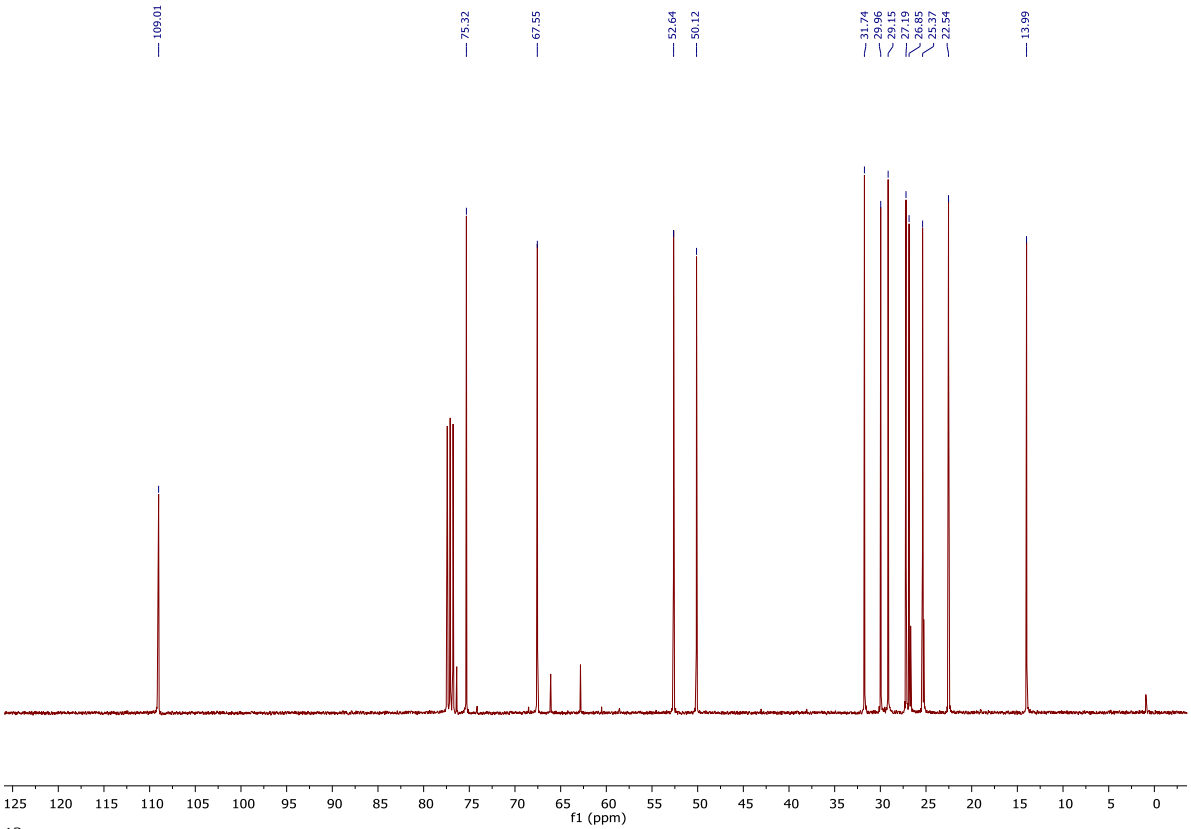


^{13}C NMR of 5-deoxy-5-(*N*-isopropyl)amino-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranoside **1b**

3) Compound **2**

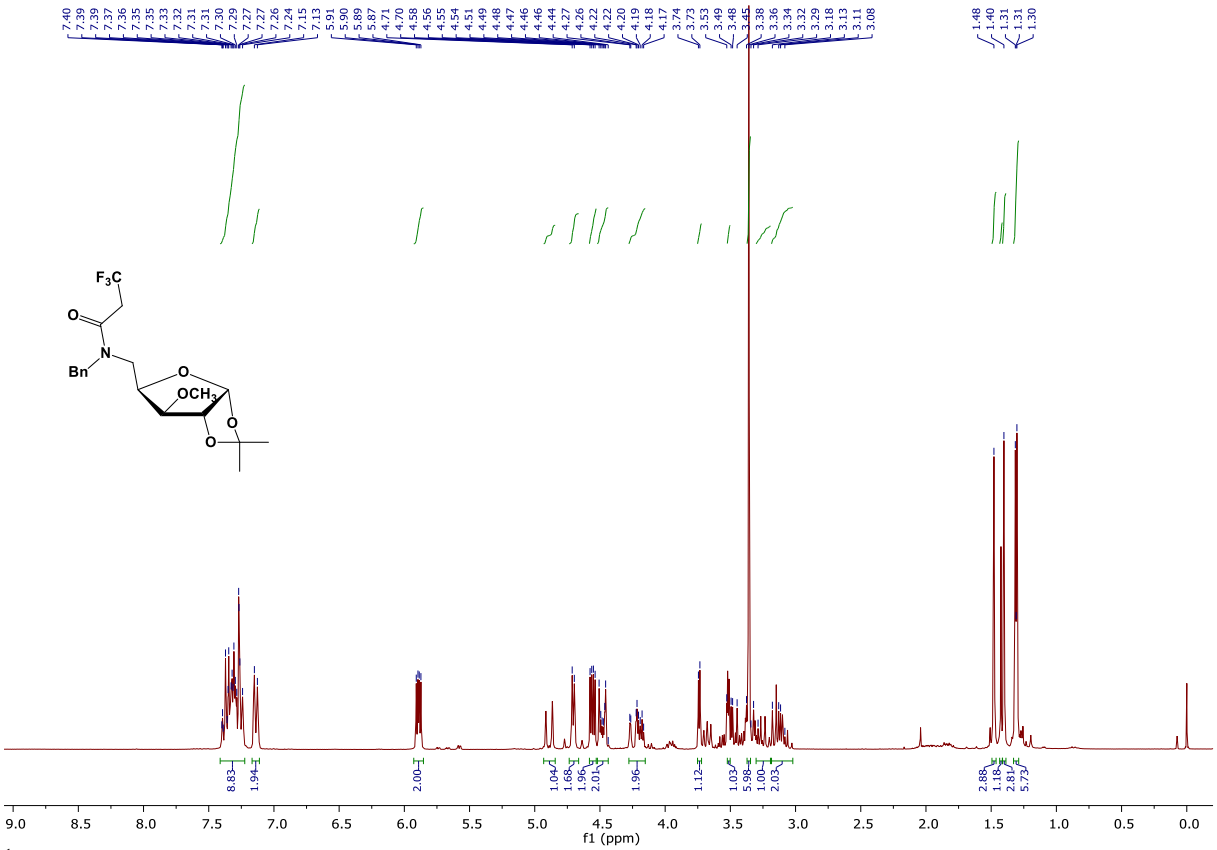


^{1}H NMR of (*S*)-*N*-((2,2-dimethyl-1,3-dioxolan-4-yl)methyl)heptan-1-amine **2**

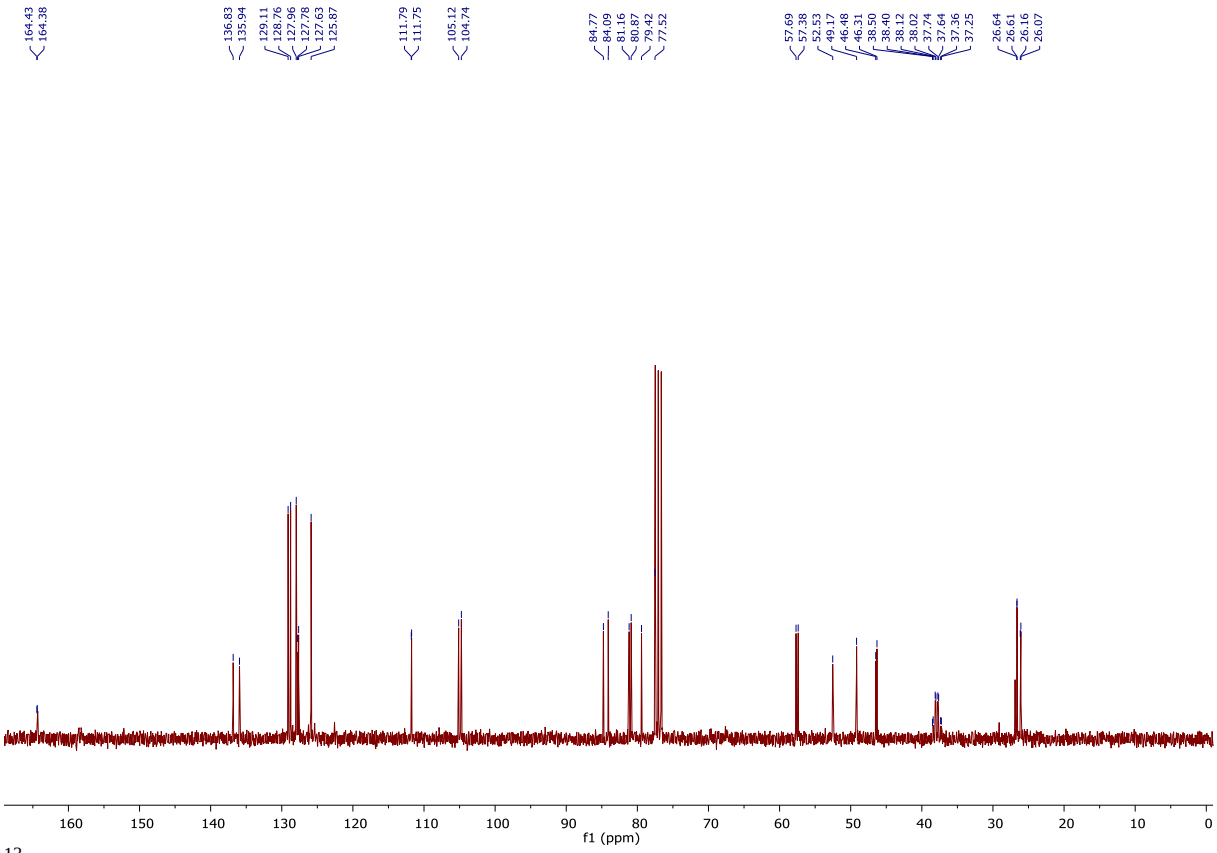


^{13}C NMR of (*S*)-*N*-((2,2-dimethyl-1,3-dioxolan-4-yl)methyl)heptan-1-amine **2**

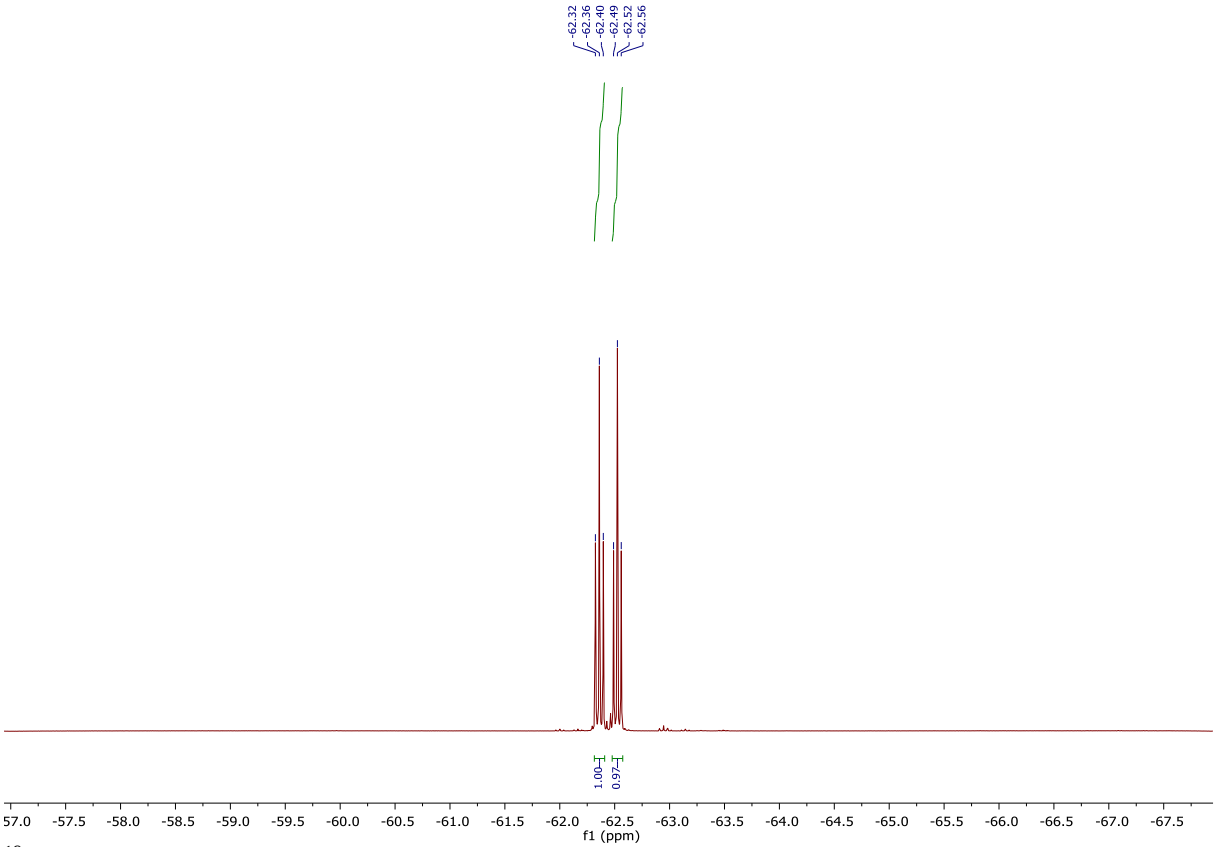
4) Compound **9a**



^{1}H NMR of *N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-3,3,3-trifluoropropanamide **9a**

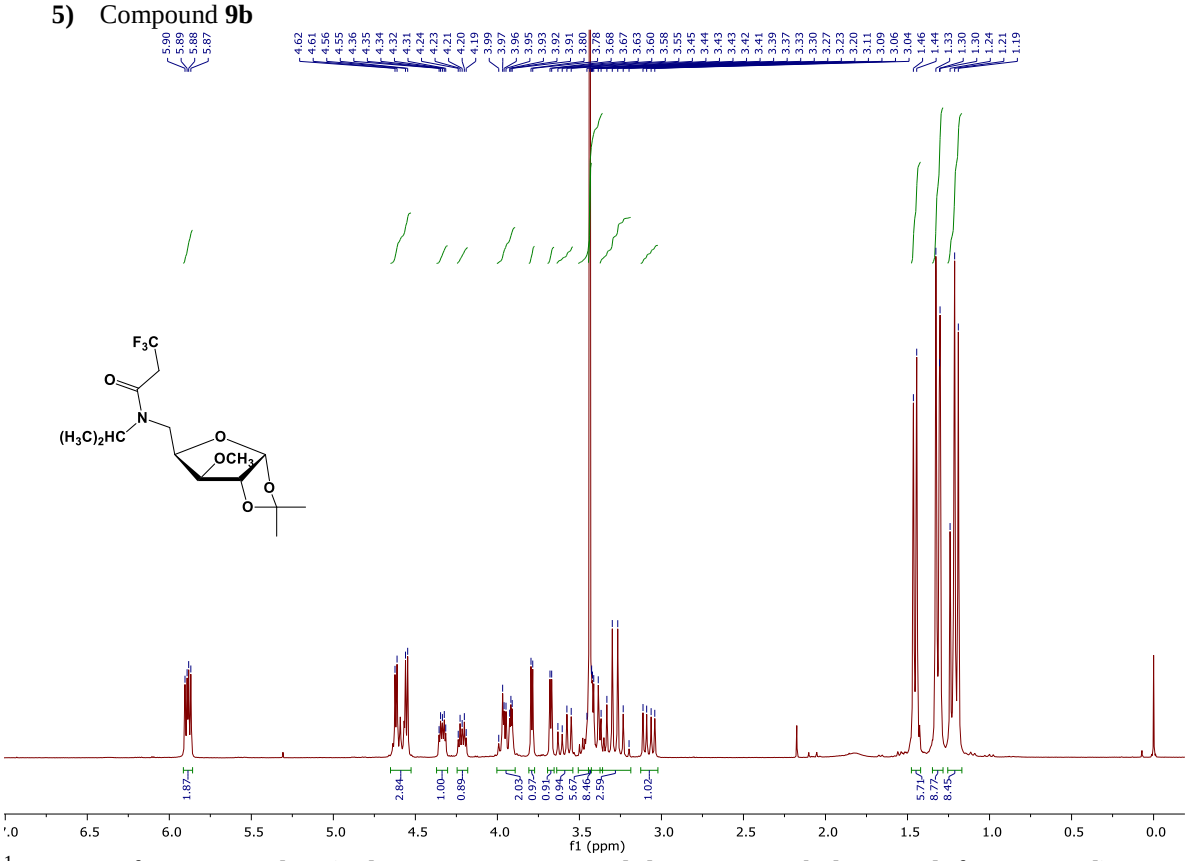


^{13}C NMR of *N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-3,3,3-trifluoropropanamide **9a**

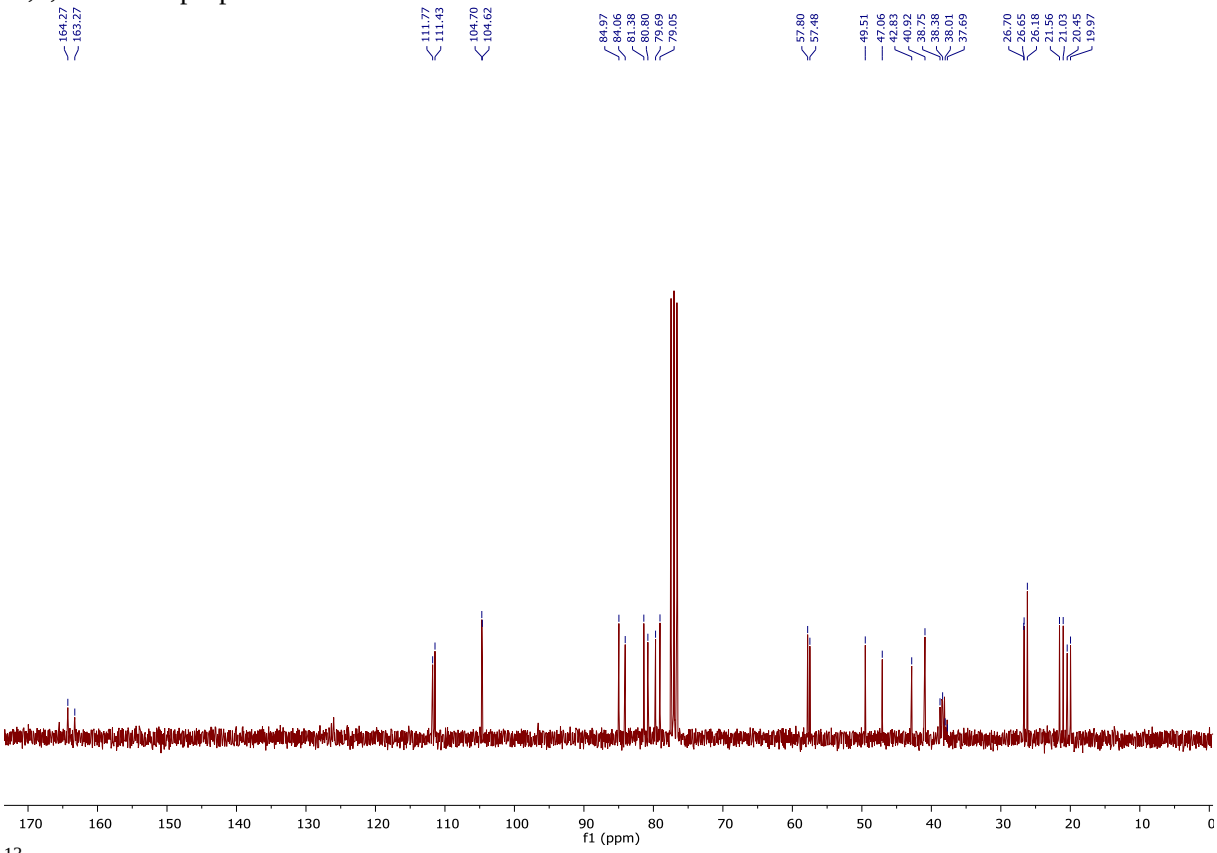


^{19}F NMR of *N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-3,3,3-trifluoropropanamide **9a**

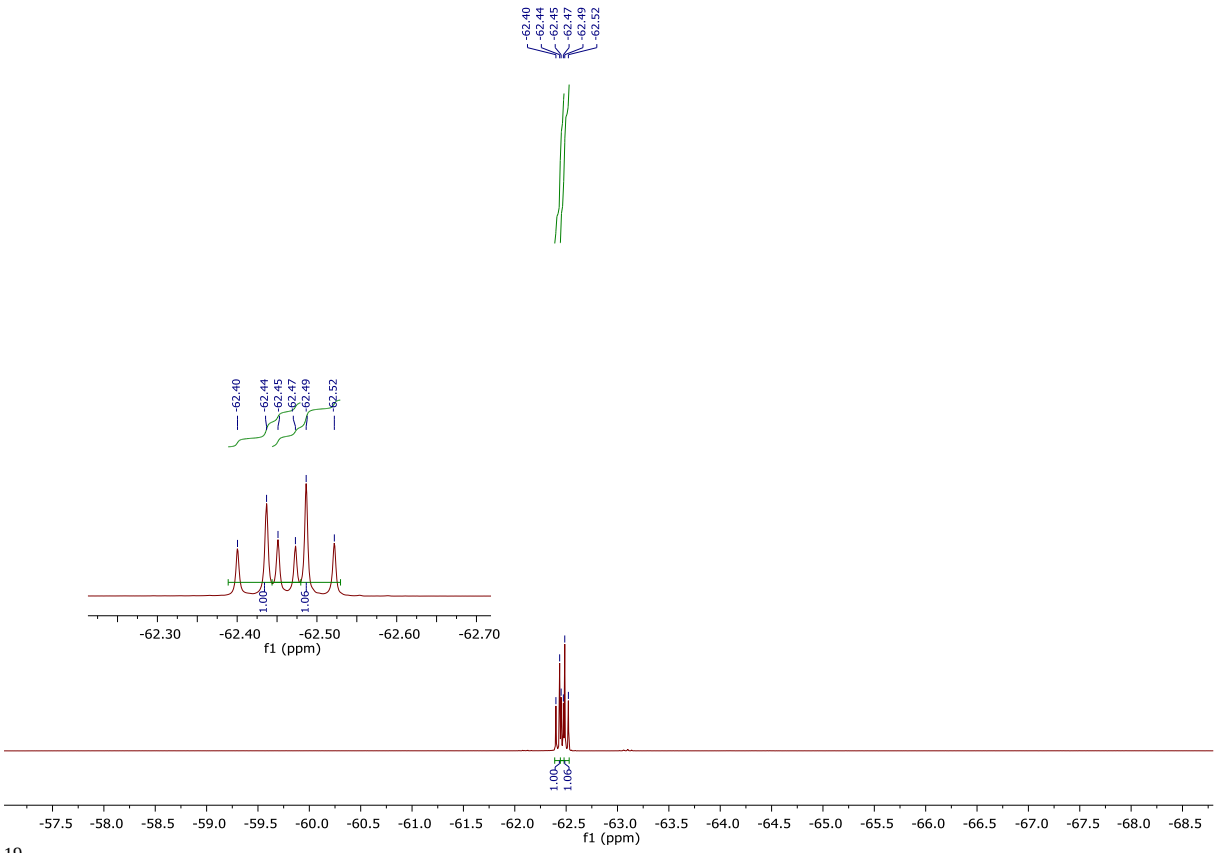
5) Compound **9b**



^{1}H NMR of *N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--3,3,3-trifluoropropanamide **9b**

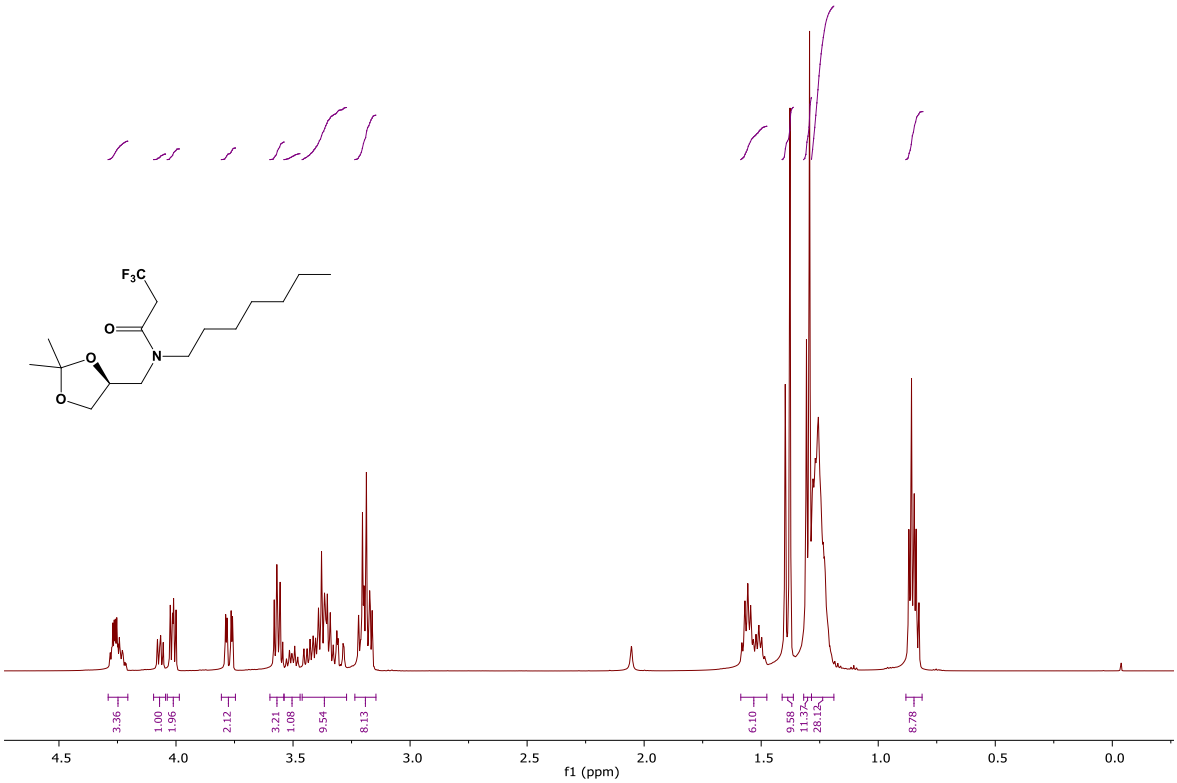


^{13}C NMR of *N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--3,3,3-trifluoropropanamide **9b**

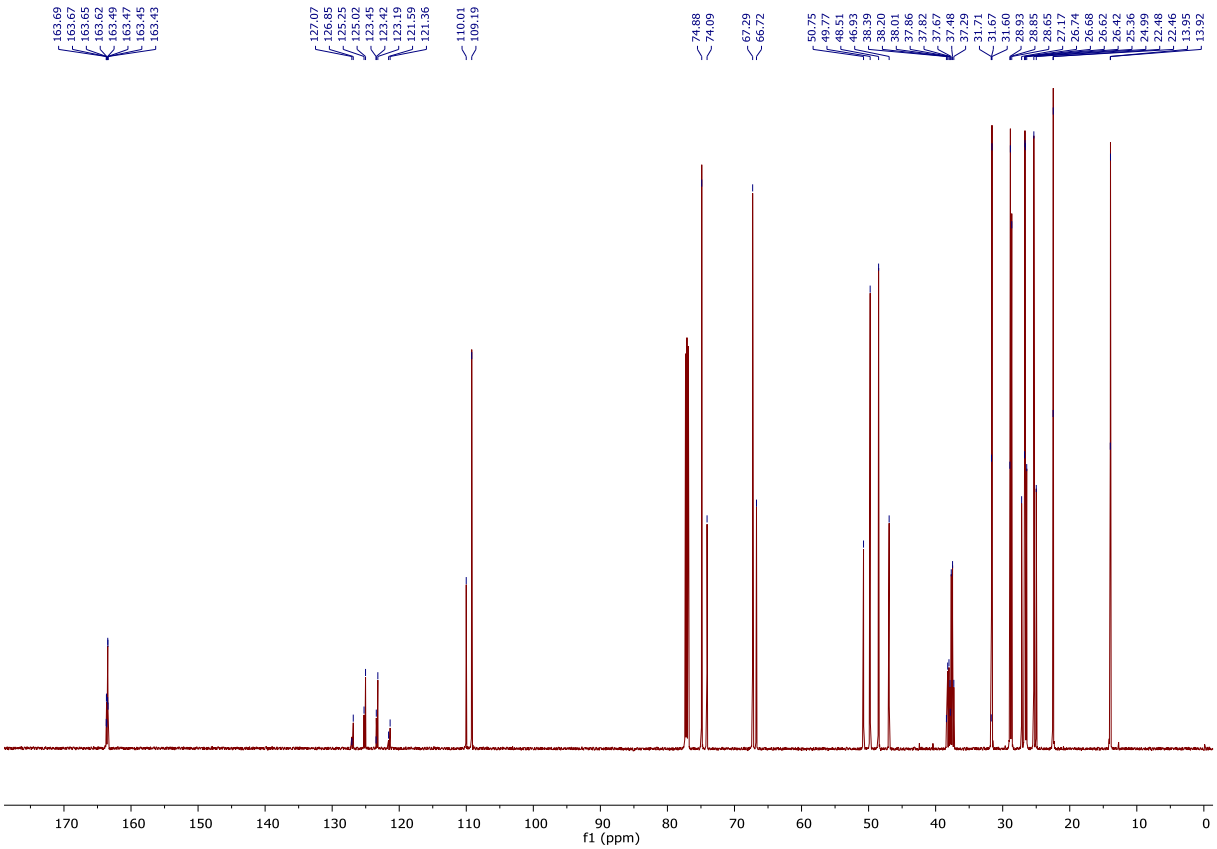


^{19}F NMR of *N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--3,3,3-trifluoropropanamide **9b**

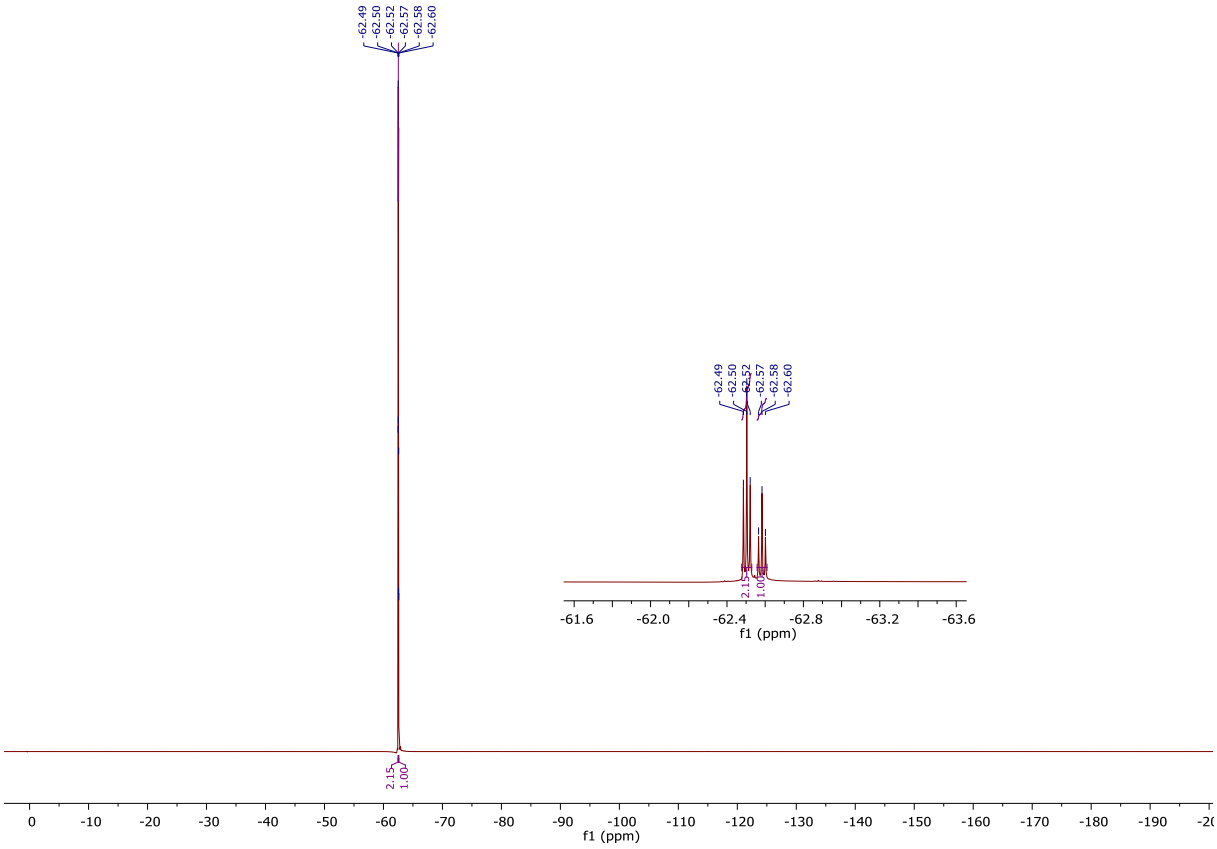
6) Compound **11**



^{1}H NMR of (*S*)-*N*-((2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-3,3,3-trifluoro-*N*-heptylpropanamide **11**

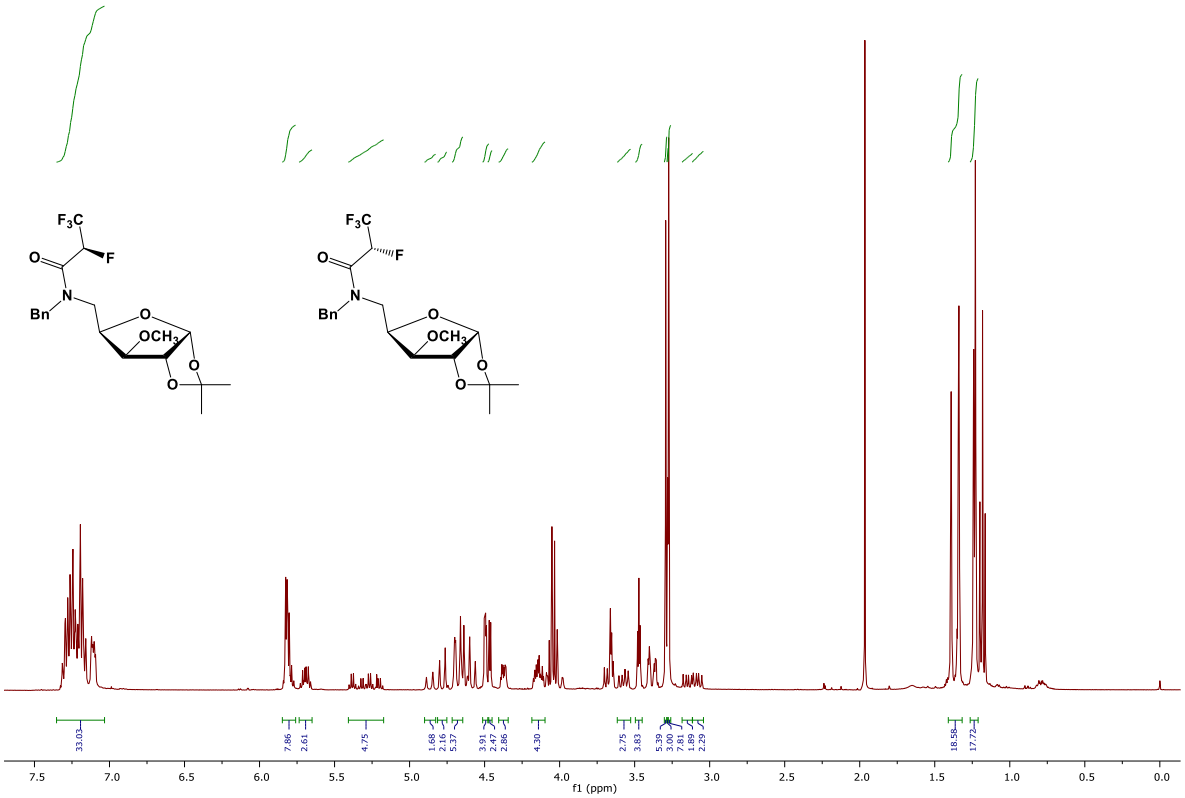


^{13}C NMR of (*S*)-*N*-((2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-3,3,3-trifluoro-*N*-heptylpropanamide **11**

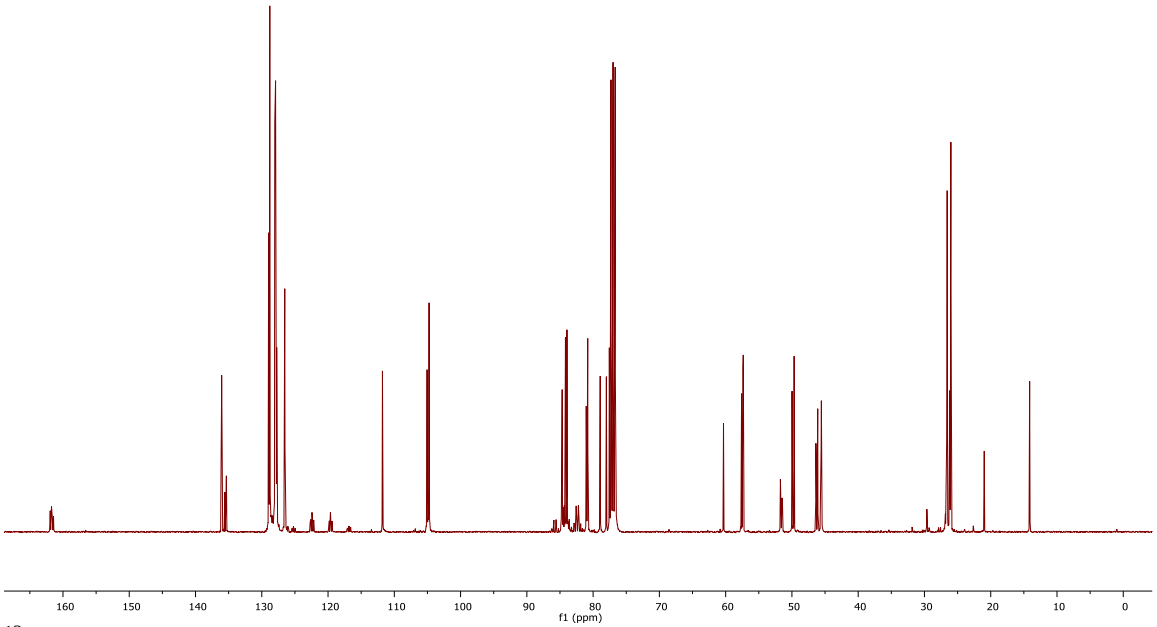


^{19}F NMR of (*S*)-*N*-((2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-3,3,3-trifluoro-*N*-heptylpropanamide **11**

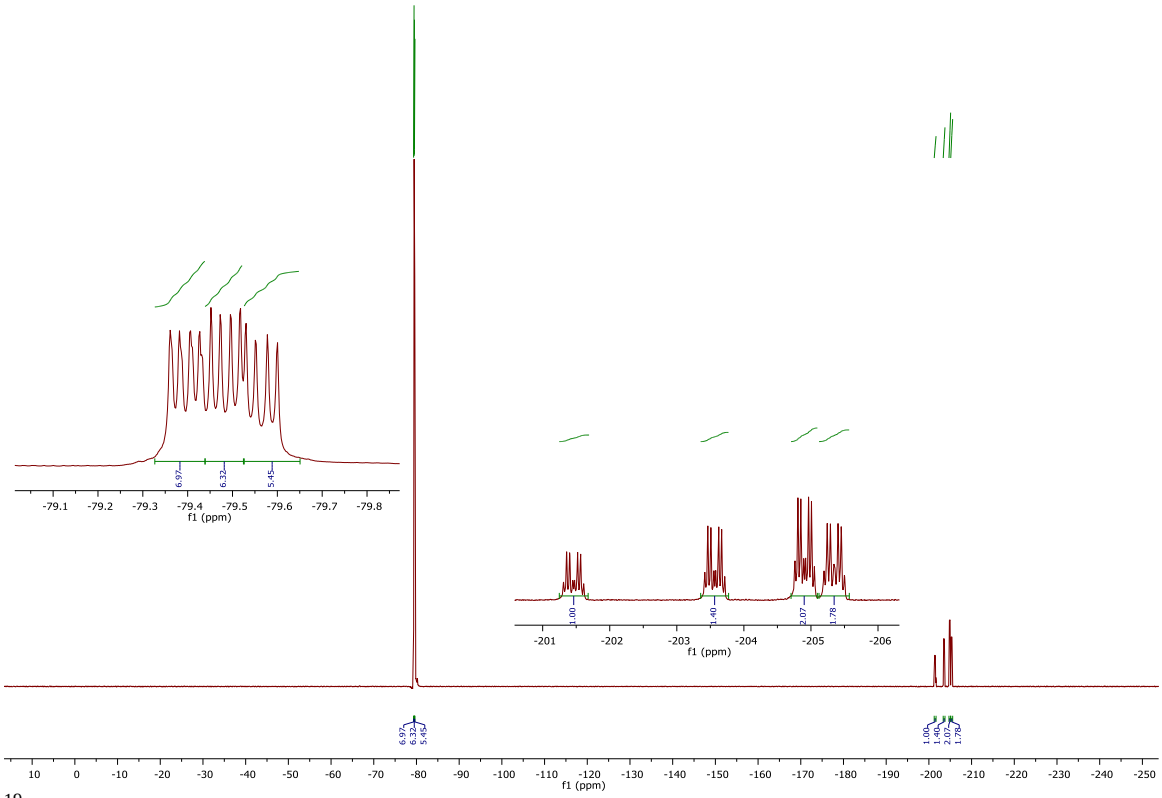
7) Compound **10a**



^{1}H NMR of (*R*/*S*)-*N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10a**

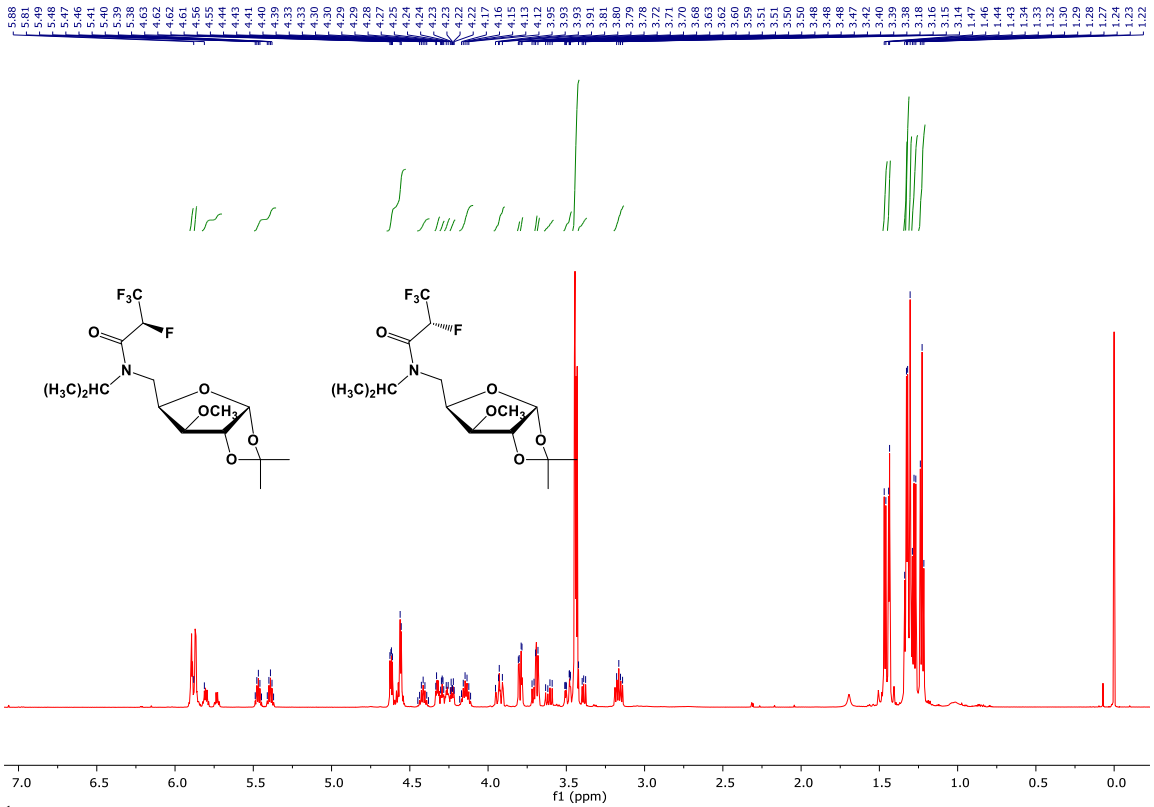


^{13}C NMR of (*R*/*S*)-*N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10a**

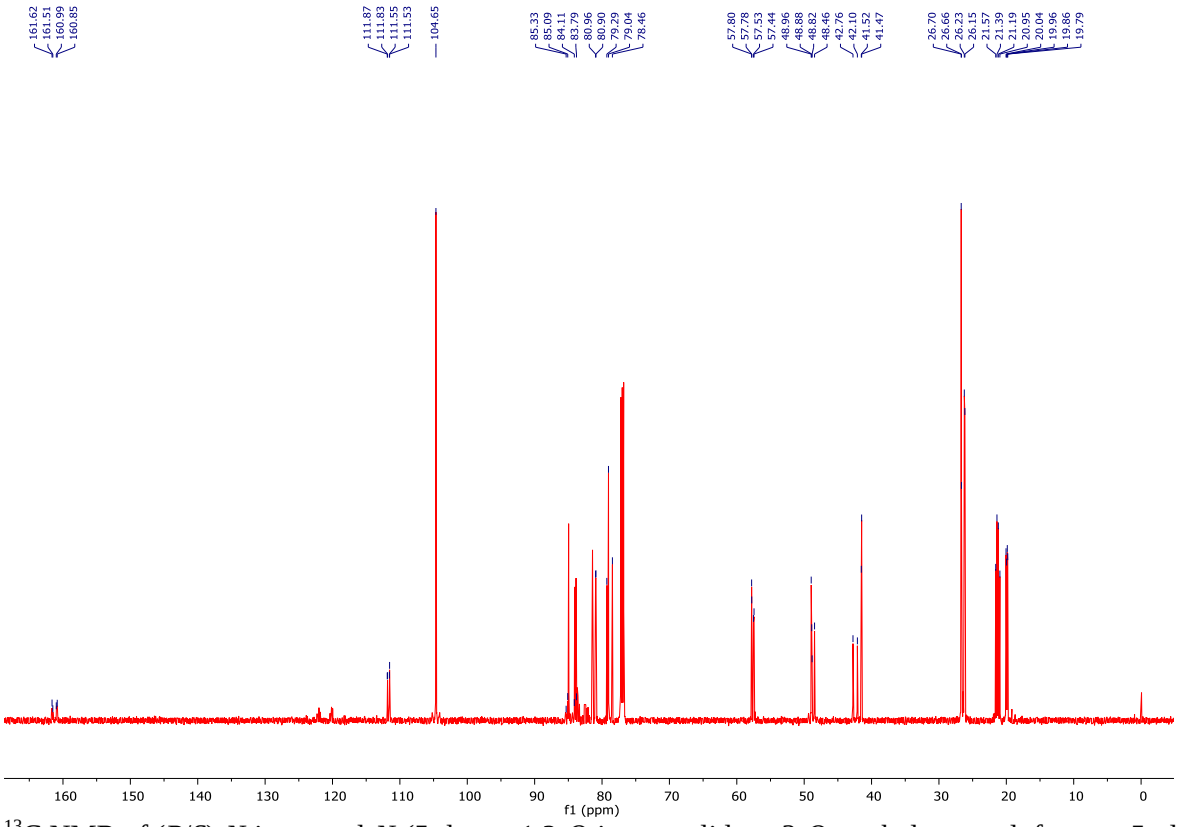


^{19}F NMR of (*R*/*S*)-*N*-benzyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10a**

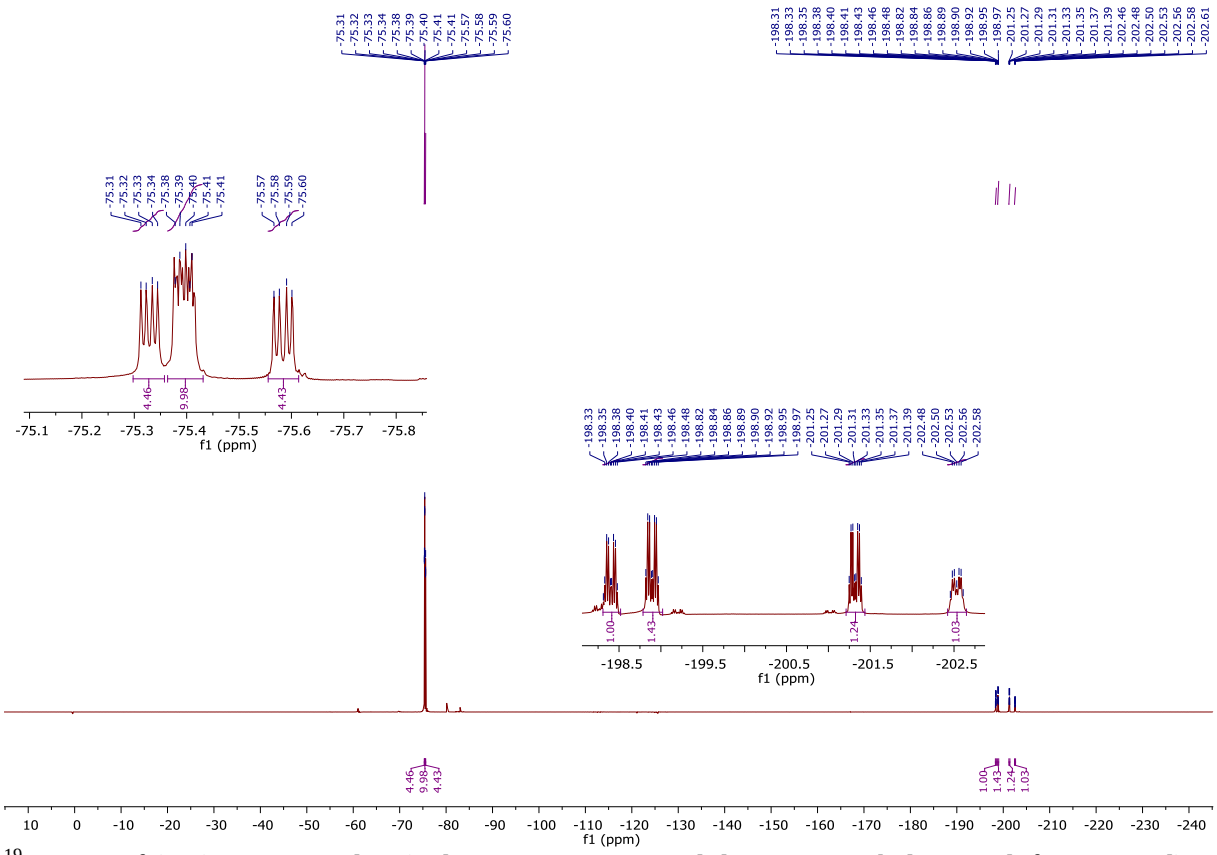
8) Compound **10b**



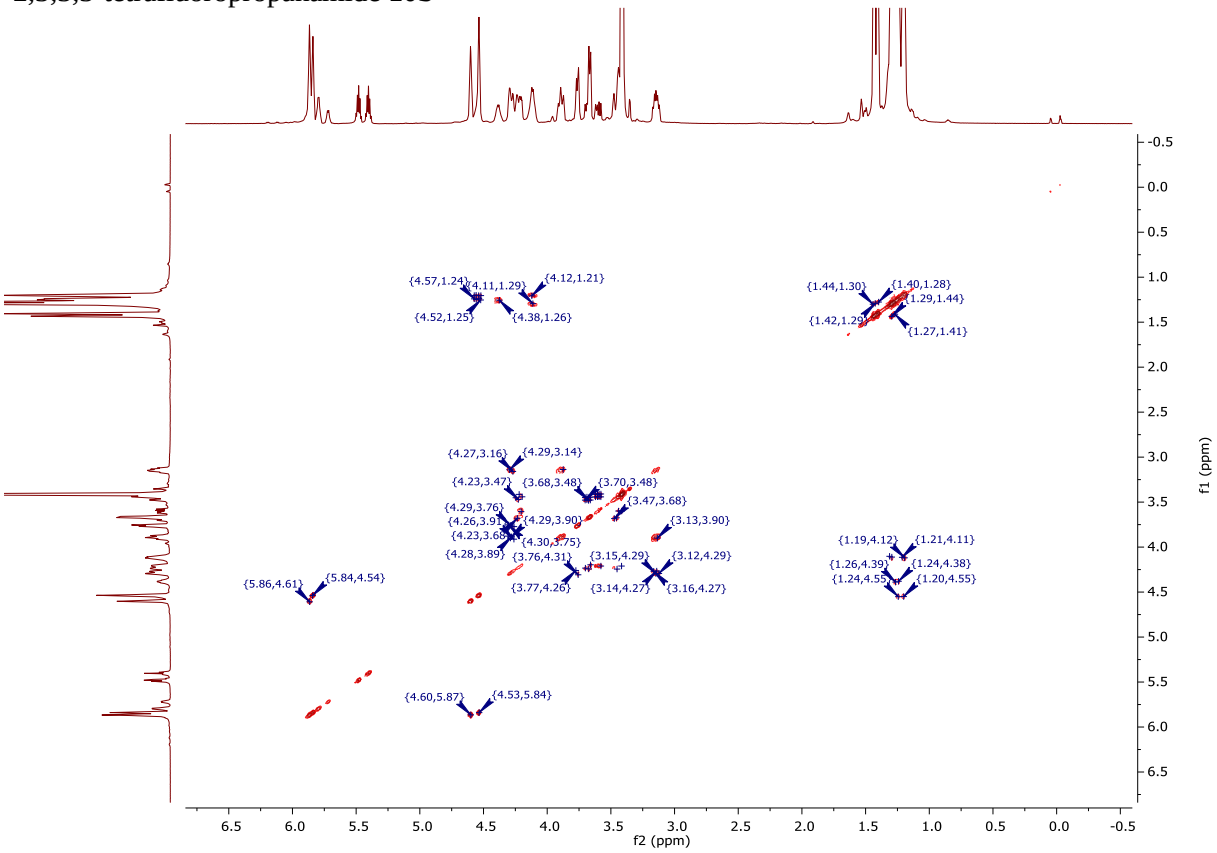
^{1}H NMR of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10b**



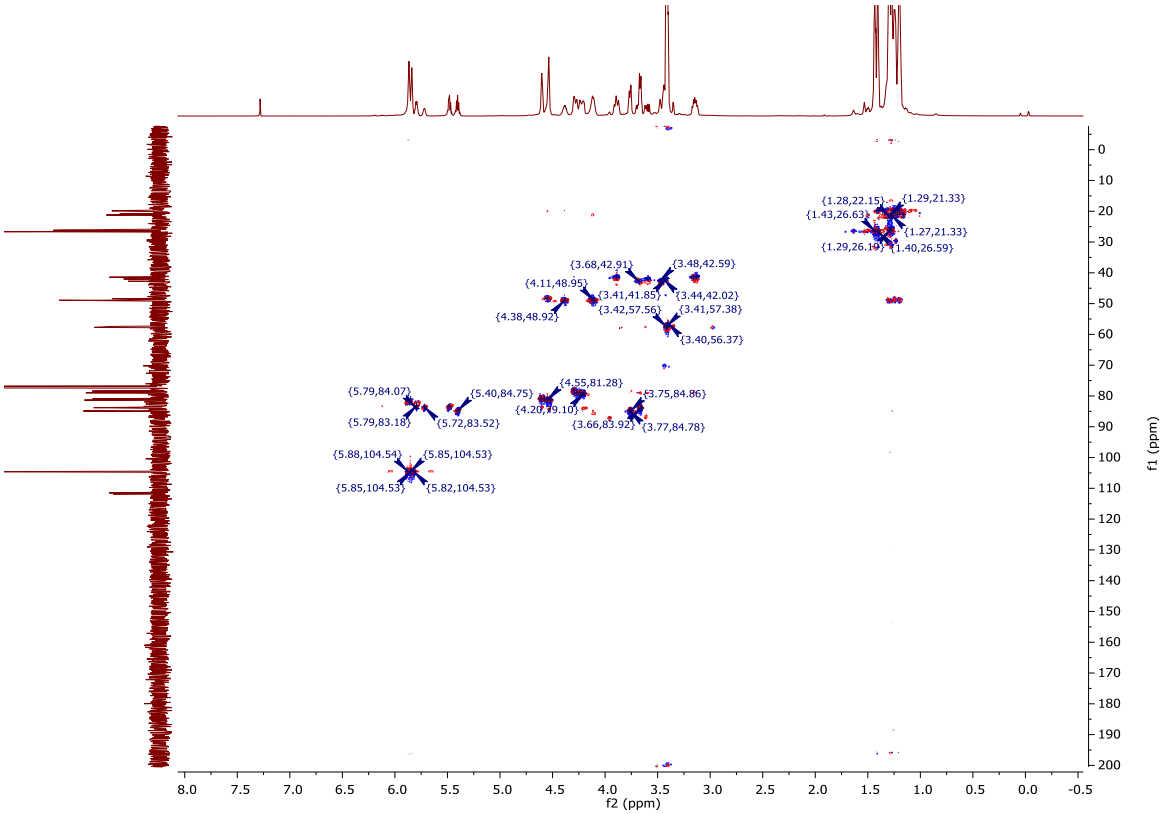
^{13}C NMR of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10b**



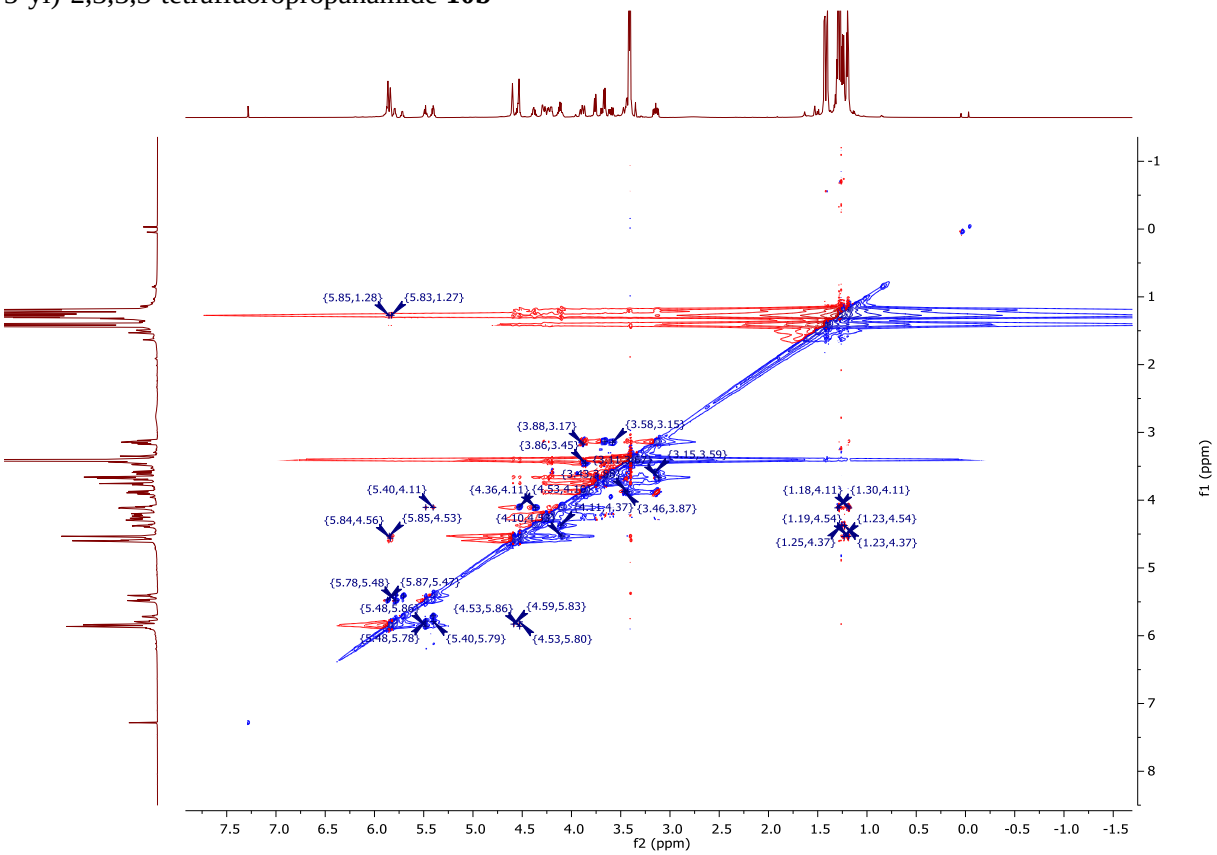
^{19}F NMR of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)--2,3,3,3-tetrafluoropropanamide **10b**



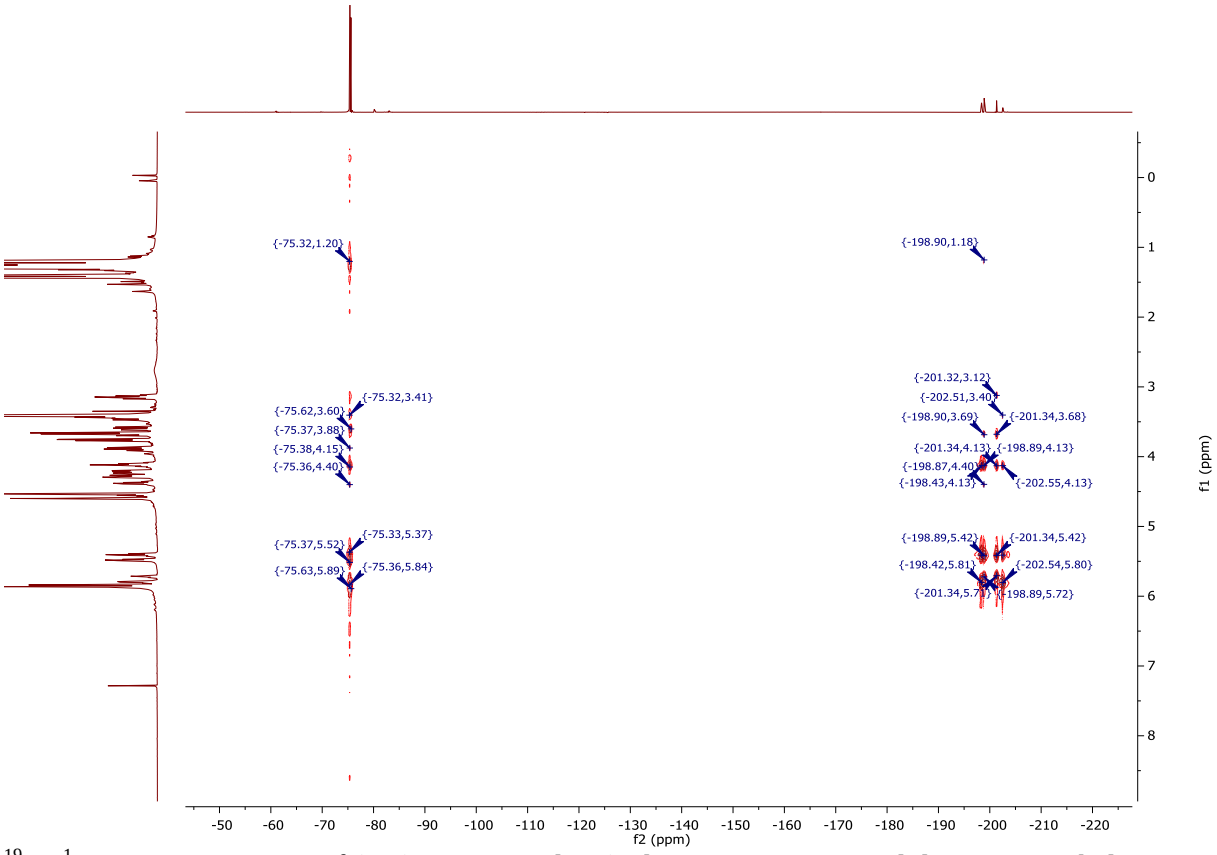
COSY spectrum of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-2,3,3,3-tetrafluoropropanamide **10b**



HSQC spectrum of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-2,3,3,3-tetrafluoropropanamide **10b**

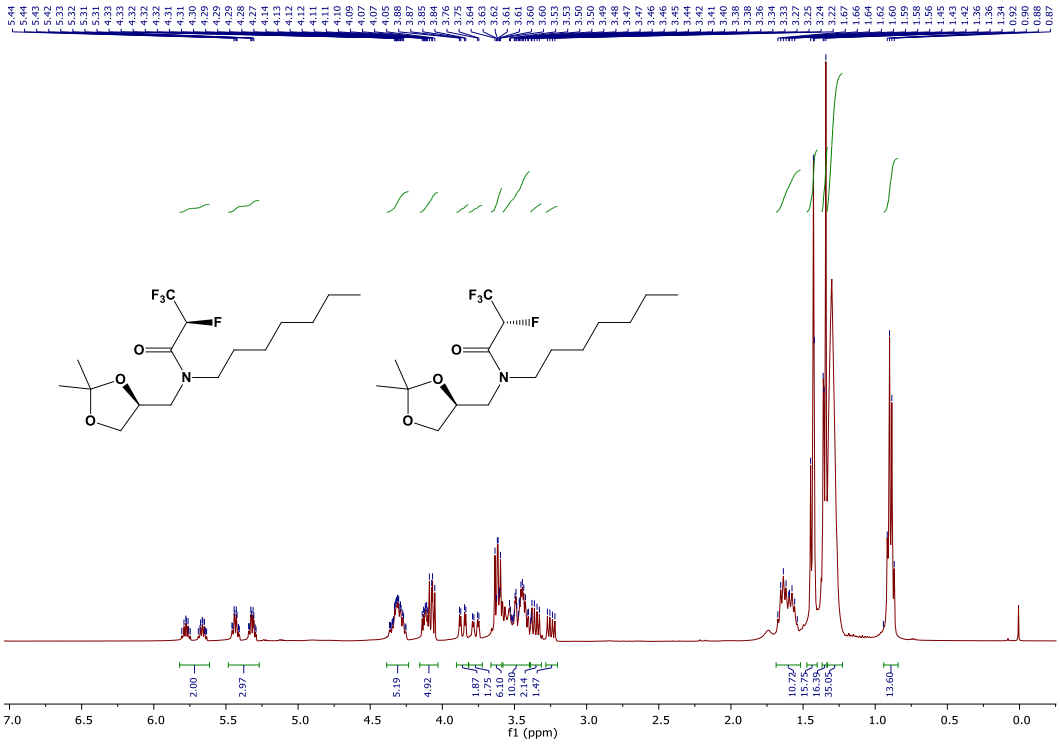


NOESY spectrum of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-2,3,3,3-tetrafluoropropanamide **10b**

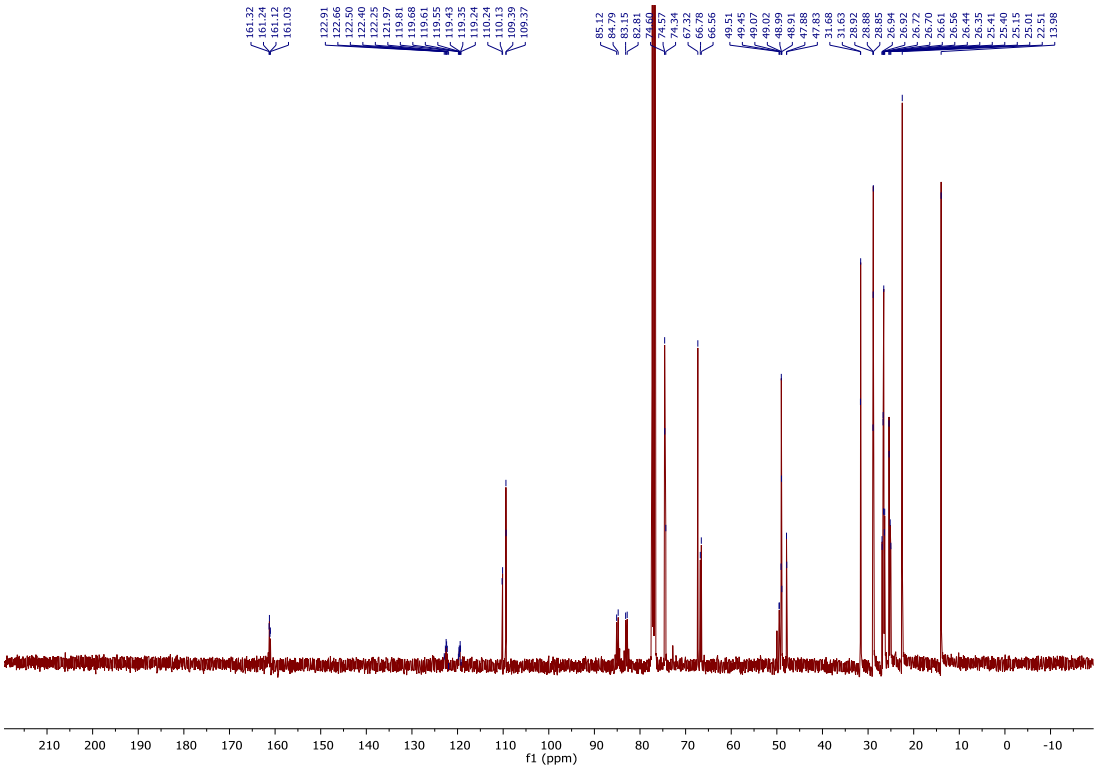


^{19}F - ^{1}H HOESY spectrum of (*R*/*S*)-*N*-isopropyl-*N*-(5-deoxy-1,2-*O*-isopropylidene-3-*O*-methyl-*α*-D-xylofuranos-5-yl)-2,3,3,3-tetrafluoropropanamide **10b**

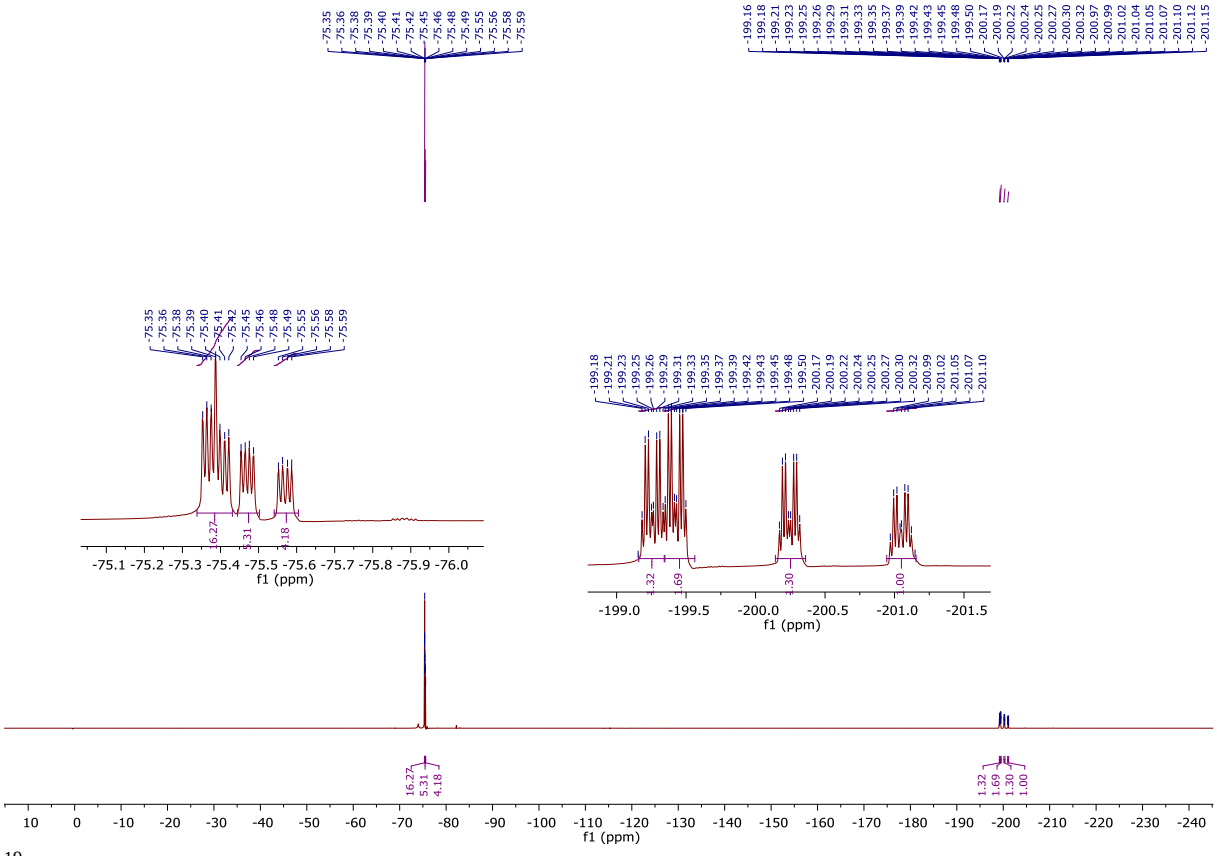
9) Compound **12**



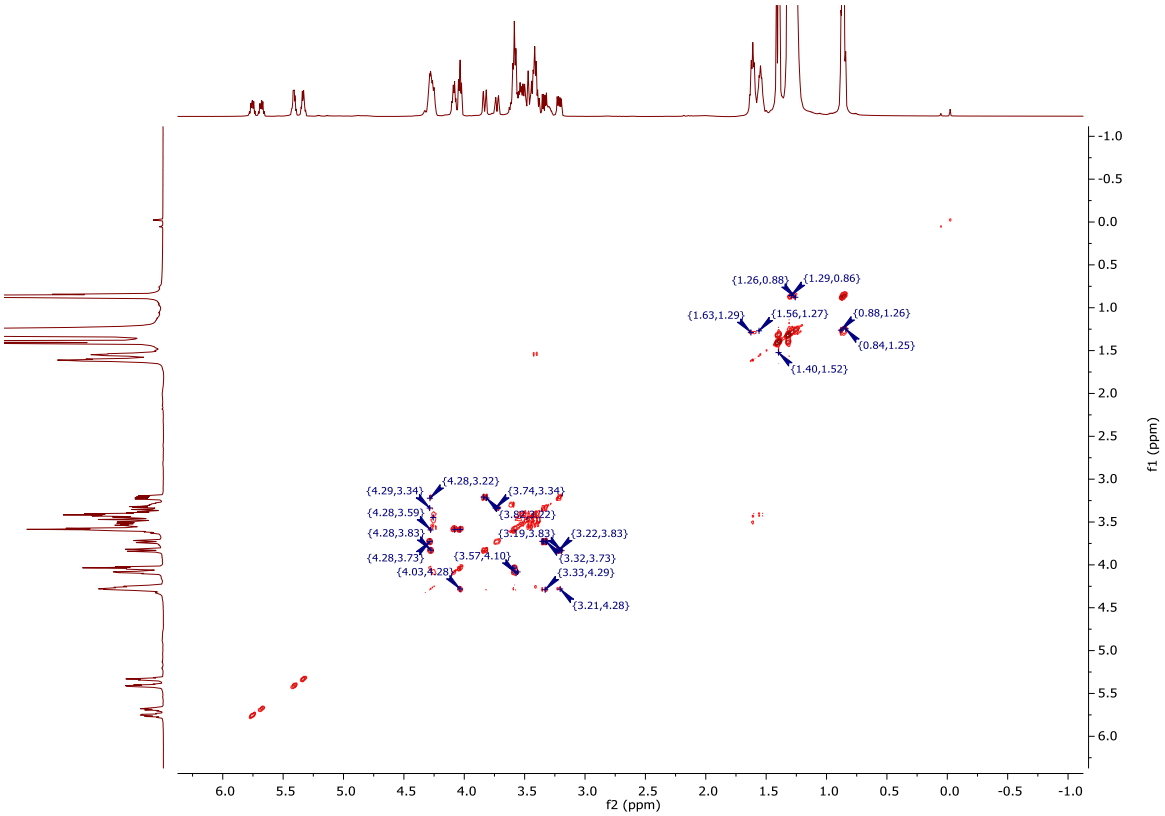
^{1}H NMR of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide**12**



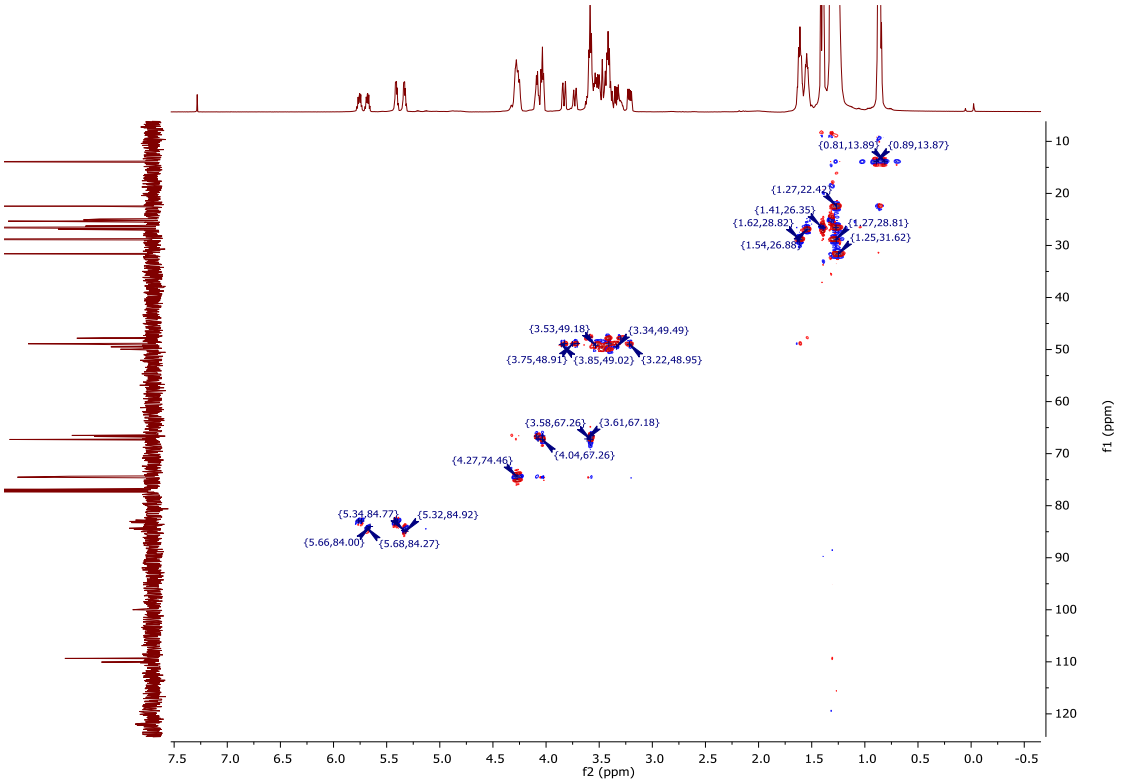
^{13}C NMR of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**



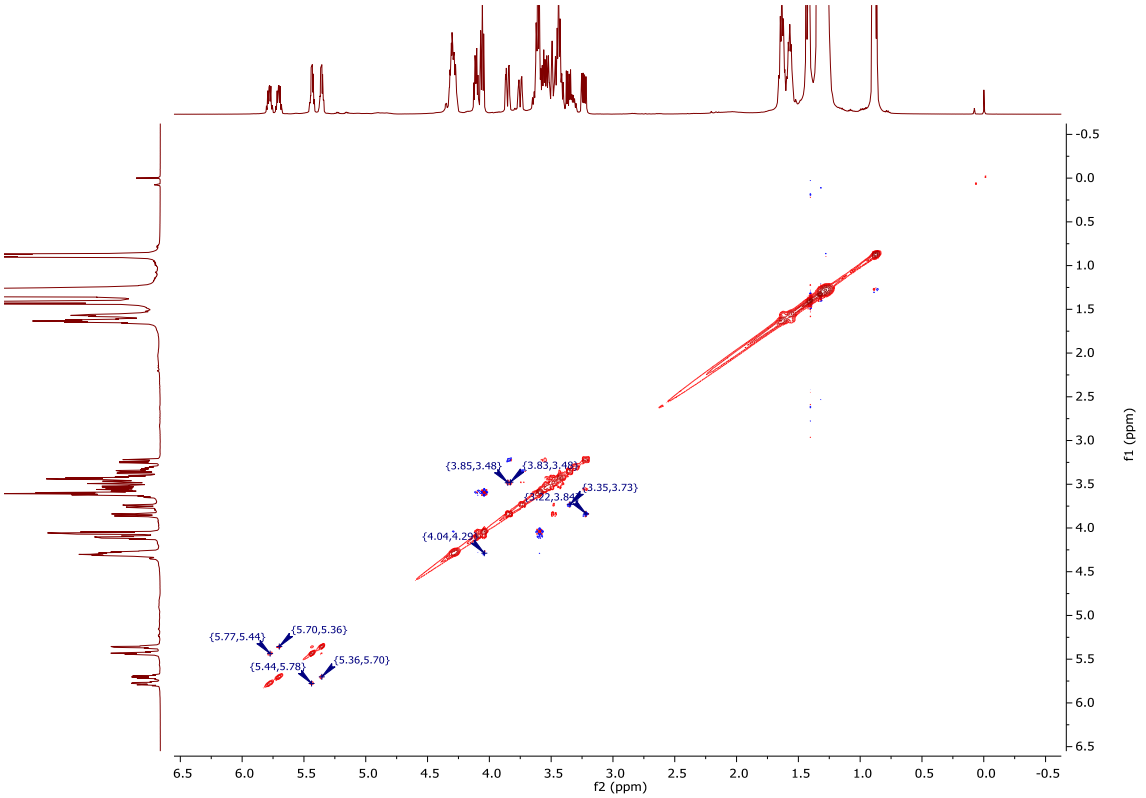
^{19}F NMR of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**



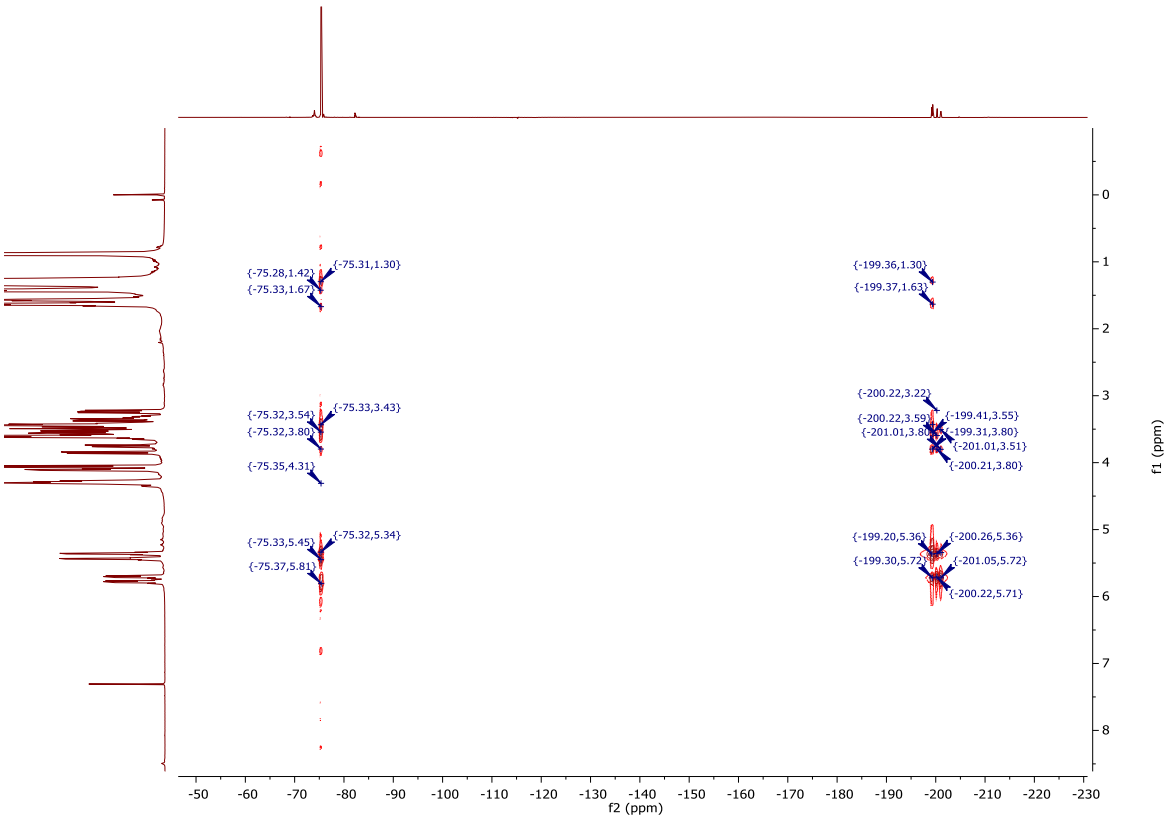
COSY spectrum of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**



HSQC spectrum of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**



NOESY spectrum of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**



^{19}F - ^{1}H HOESY spectrum of (*R*/*S*)-*N*-(((*S*)-2,2-dimethyl-1,3-dioxolan-4-yl)methyl)-2,3,3,3-tetrafluoro-*N*-heptylpropanamide **12**

II Crystallographic and Experimental Data for 10a at 295 K

Compound	**10a**
Formula	$C_{19}H_{23}F_4NO_5$
Temperature (K)	295(2)
Formula weight	421.38
Crystal colour	Colourless
Crystal size (mm)	0.12x0.05x0.03
Crystal system	Triclinic
Space group	*P*1
Unit cell parameters (Å/°)	
a	6.0766(4)
b	9.4360(3)
c	9.7318(4)
α	73.389(3)
β	72.298(4)
γ	84.890(4)
Volume (Å^3)	509.41(4)
Z	1
D_x (g cm^{-3})	1.374
Radiation source	CuK_α
Wavelength λ (Å)	1.54184
Absorption coefficient (mm^{-1})	1.062
F(000) (e)	220
2θ min/max (^{o})	4.89/74.49
Min./Max. indices	
h	-7/6
k	-11/11
l	-12/12
Reflections collected/unique	12459/4002
R_{int}	0.0222
Observed reflections (I>4σ$_I$)	3480
Data/parameters	4002/251
Goodness of fit on F^2	1.139
Final R_1/wR_2 indices (I>4σ$_I$)	0.0782/ 0.2411
R_1/wR_2 indices (all data)	0.0838/0.2534
$\Delta\sigma_{max}$, $\Delta\sigma_{min}$ (eÅ^{-3})	0.690, -0.585
Weighting scheme[a]: x; y	0.183, 0.034
Extinction coefficient	–
Absorption correction type	multi-scan
Sample transmission min/max	0.85/ 1.00

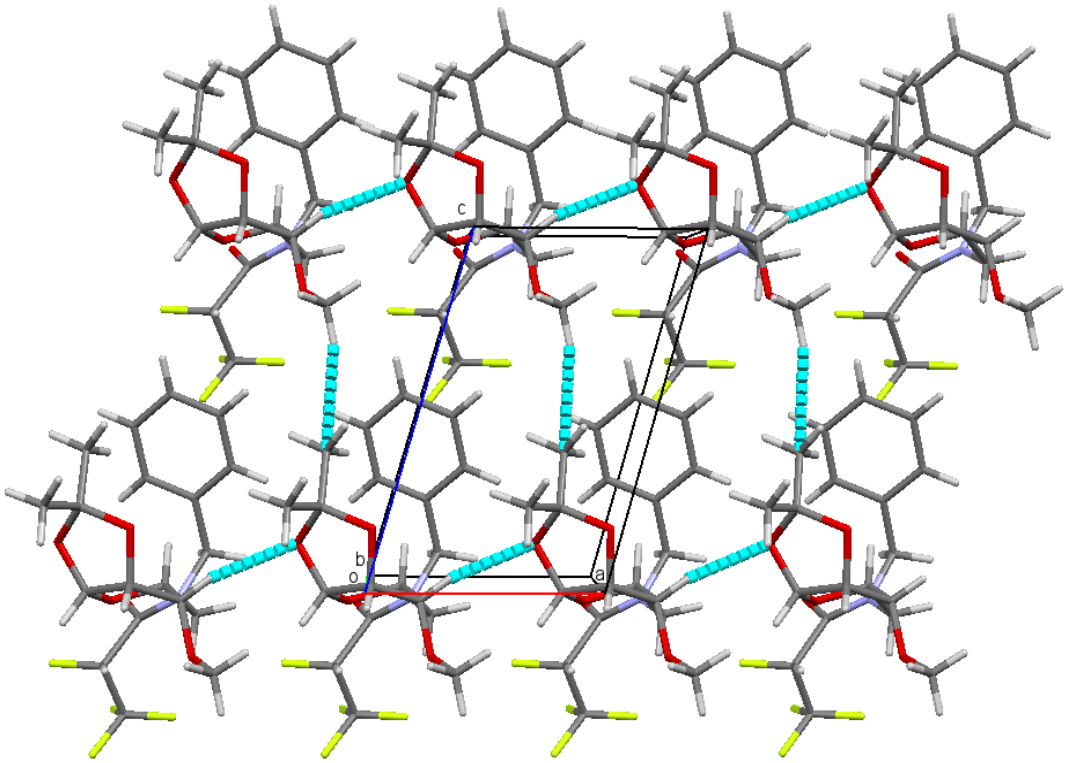


Figure 1. Autostereographic projection [1] of the **10a** structure, with short intermolecular contacts CH···O and CH···CH_3 indicated by cyan dotted lines.

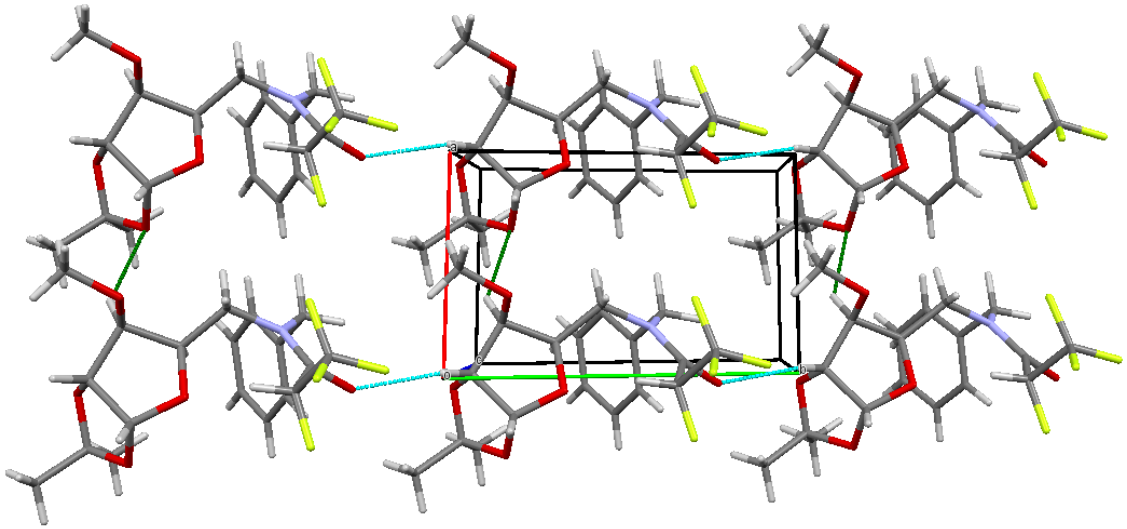


Figure 2. Autostereographic projection [1] of the **10a** structure, with short intermolecular contacts C13H13···O3 and C14H14···O1 indicated by green and cyan dotted lines, respectively.

Reference

[1] Katrusiak A., Crystallographic Autostereograms, *J. Mol. Graph. Model.* **2001**, *19*(3-4), 363-367